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Determination Some Structural Characteristic of the Compound $NiSnO_3$

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Abstract
Nickel stanate ($NiSnO_3$) was synthesized by a chemical precipitation method. The synthesized samples were characterized using X-ray powder diffraction technology (XRD). The temperature of synthesis was 700°C. Muller indexes (hkl) were calculated for the production, and it was clear that , the compound crystallized according to hexagonal lattice with following parameters a=5.1879 (A^0), c=14.2209 (A^0),
V= 331.4777(A^0)^3. The space group of symmetry is R3c. The IR spectroscopy encourage our results during the bonding vibrations of Zn-O, Ni-O.The thermal characteristic shows four endothermic effects.
Keywords: crystal structure, mixed oxides, Nickel stanate.

1. Introduction

The inorganic compounds that have a structure of multiple oxides are one of the very important elements in industry, which has attracted much attention recently because of their large-scale applications in various areas of life, such as used in sensors for chemical gases such as ethanol gas, and in the detection of moisture devices, and It is used in many electronic devices and entered into a manufacturing ceramic materials and thin film sensitive devices [1-2].

In our present study, we examined mixed oxides-style –the so called Perovskites ,which take the general chemical formula ABO_3 where A is a divalent or monovalent metal and B is a tetra- or pentavalent atom [3].

Many researchers have cared for manufacturing various Nickel stanate have been several methods to adopt in order to obtain the oxide of the most important of these methods: the way of common deposition and method of solid-state and the way the Sol-Gel and the method of heat treatment hydrothermal and other methods.

The method of co-deposition is one of the important ways to which the attention of scientists in the recent period has paid, and this trend attributed to the possibility by which we obtain high-purity compounds and we get on with great homogeneity and small crystal sizes out of solvents, salts metals embedded installed oxides mixed required, this method does not require high temperatures for the synthesis and the factors affecting the outcome of this method is the reaction temperature , the degree of pH , the stirring speed , and time of the deposition. However, the disadvantages of this method it is possible that we will get more of the process when using the ratio molar is appropriate the synthesis furthermore, the solubility of the salts used should be close.

Recently, ternary $NiSnO_3$ was first reported as an anode for Li-ion batteries, presenting superior electrochemical performance to that of the NiO/SnO_2 mixture. This was proposed to be due to the 'self-matrix' function of the discharge products that enables buffering the volume change and preventing the aggregation upon cycling. In this article, direct evidence of the 'self-matrix' mechanism of $NiSnO_3$ has been observed via ex situ transmission electron microscopy (TEM) and selected area electron diffraction (SAED). [4]

In one of the articles was a compound nickel Stanat prepare the following way. A mixture of NiO and SnO_2 (marked NAS) was synthesized by a polymerized complex technique. In a typical procedure, tin(II) acetate ($Sn(CH_3COO)_2$, 0.1 mol) was dissolved in the solvent of ethylene glycol (0.4 mol), and subsequently anhydrous citric acid (0.1 mol) was added to the obtained solution and fully dissolved at 50 °C for 3 h. Next nickel(II) acetate ($Ni(CH3COO)_2$, 0.1 mol) was added and the resultant mixture was stirred at 80 °C for 6 h. After that, the obtained transparent solution was heated at 135 °C for 48 h to remove the excess solvent and to promote polymerization.

Obtained powder was heated at 350 °C for 5 h, and then at 600 °C for 10 h in the muffle furnace. In comparison, commercial nanoscaled nickel tin oxide ($NiSnO_3$, NSO) powders was dried at 100 °C for 72 h under vacuum, and then used without further purification. [5]

2. The goal of the research:

The synthesis of Nickel stanate way joint precipitation from Nickel chloride quaternary salt and Tin chloride, and the study of crystalline and structural characteristics in general, and to find the temperature of synthesis and the appropriate conditions for it, such as pH center and focus of aqueous solutions of metal chlorides entering the process.

3. Necessary to conduct research materials:

High purity materials were used for analytical goals:

- Tin tetrachloride (98.0%) $SnCl_4.5H2O$
- Nickel chloride (98.0%) $NiCl_2.6H_2O$
- Ammonium hydroxide NH_4OH

4. Practical section:

4.1. Sample preparation and method of synthesis:

In our research we have adopted this method of co-deposition in order to obtain the required compounds to search. The weights of the raw materials used in account out of the molar weights of raw materials according to the following equation:

$$SnCl_4.5H_2O + NiCl_2.6H_2O + 6NH_3H_2O \rightarrow NiSnO_3 + 6NH_4Cl + 14H_2O$$

And Table 1 shows the weights of the raw materials used and calculated in accordance with the previous equation, and weights required were calculated on the basis that the composite output desired amount equal to 5gr. Table shows size of distilled water to be affixed in accordance with the solubility of salt for the preparation of solutions according to the concentrations required.

	salt	
	$NiCl_2.6H_2O$	$SnCl_4.5H_2O$
concentration of brine (M)	0.8M	0.6M
salt mass(gr)	4.3045	6.3484
added distilled water volume (ml)	22.635≈ 23	30.180≈30

The preparation of Nickel chloride (II) solution concentration Molara of (0.8 M) and preheat the degree 65°C, and then was added quantity calculated accurately from the ammonium hydroxide solution and slowly and gradually in batches as depositing and adjusted for the value of the pH the same time, and the addition continues until the arrival of the value of the pH to moderate value when (pH = 7), and in the second step is the preparation of quaternary tin chloride solution $SnCl_4.5H2O$ concentration (0.6M) and was added slowly to the mix maintaining the pH value at the value 7 using ammonium hydroxide, where it was to maintain the solution temperature throughout artificiality period when the fixed rate 65°C with rapid and constant stirring. After completion of the addition the whole tin chloride solution and steadily the value of pH at 7, we left the solution exposed to heat up at the degree 65°C with rapid stirring for two hours, in order to get a larger percentage of homogeneity output of the deposit, and then the mixture was left to settle down and start the deposition process for a full day at room temperature, then the resulting sludge was precipitated and washed several times with distilled water filtration, to get rid of excess chlorine ions, and dried for two hours at 105°C degree.

The second phase of the work included putting in the dried sample crucibles in durable ceramic high temperature of about 1200°C in order to burn in different temperatures starting from

550°C until 1000°C, taking care to observe the process through getting the x-ray diffraction diagram at different temperatures, and comparing the resulting charts with x-ray diffraction initial compounds schemes. Incineration has continued at each temperature for a period of time ranging from 6 hours, to see the best degree for the synthesis; that degree of heat that indicates the emergence of a single phase (single pure compound) and is therefore the desired compound in accordance with molar ratio taken.

4. 2. Results and discussion:

4.2.1 Instant analysis of X-ray: The Nickel stanate Crematorium analysis at different temperatures by x-ray diffraction device X-Ray Powder Diffractometer from a company (Philips-PW-1840).

Figure 2. shows the XRD pattern of $NiSnO_3$ for 1:1 weight ratio, which prepared by a chemical precipitation method and annealed at 650°C for 6 hours. It has been calculating the distance between the crystalline levels (d) by the value of the diffraction angles by Prague relationship $\mathbf{n\lambda = 2d \sin\theta}$. Then was Muller indexes (hkl) expense following the method of trial and error and starting to take shape patterns premise of Top analogy to the minimum.

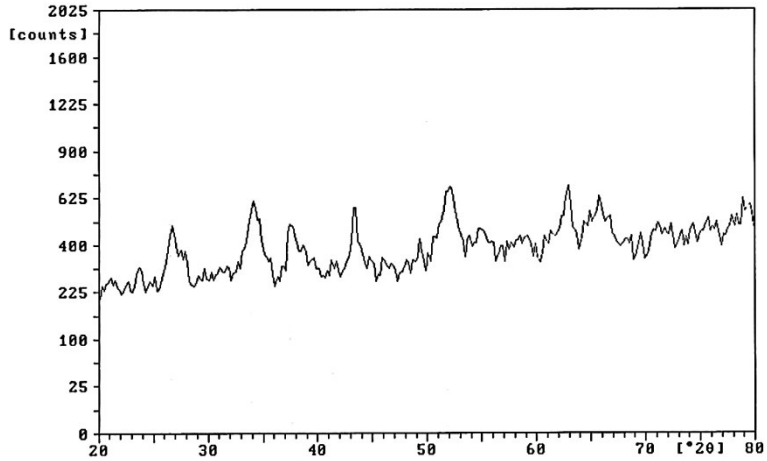


Figure 1. x-ray diffraction of the incomplete output
in wholesale ZnO-SnO_2 scheme (T = 650°C)

Note from the former diagram the appearance of some peaks belonging to the desired compound was manufactured with residue tops (low intensities) of raw materials which are oxides. These peaks belonging to preliminary oxides decrease with follow-up synthesis process and the desired compound tops look more severe until we get to the synthesis of a single phase.

The following chart shows the yaw values Nickel stanate compound prepared in a manner common deposition and that resulted when the follow-up increase in temperature reached up to 700°C, and has been fixed for six hours, see Figure 2.

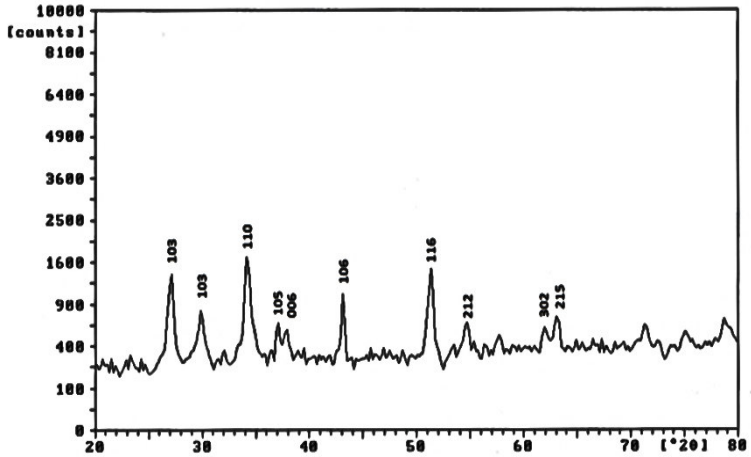


Figure 2. x-ray diffraction of the compound
resulting $NiSnO_3$ scheme (T = 700°C)

The peaks of XRD pattern of the sample was analyzed by matching with standard JCPDS No.: 052-1381 data file. At the end of the current calculations we notice that the compound corresponds to the pattern of hexagonal symmetry and all the Muller indexes (hkl) values for all the peaks are consistent with this pattern of crystallization, which is consistent with the following relationship

$$\frac{1}{d^2} = \frac{4}{3}\frac{h^2 + k^2 + hk}{a^2} + \frac{l^2}{c^2}$$

The average was calculated for each of the static lattice constants a and c:

a=5.1879 (A^0), c=14.2209 (A^0),

the basic cell size was calculated according to hexagon pattern crystallisation of the following relationship V = $a^2c \sin(60°)$. $(A°)^3$

And the following value gave the size of the studied cell V= 331.4777$(A^0)^3$.then experimental density of the material resulting in a manner flask density (picknometer) was measured for three times in a row and took the average value for them, which amounted to: ρt = 6.7726 gr/cm^3. Depending on the material's density number of formulas in a single crystalline cell was calculated according to the following relationship:

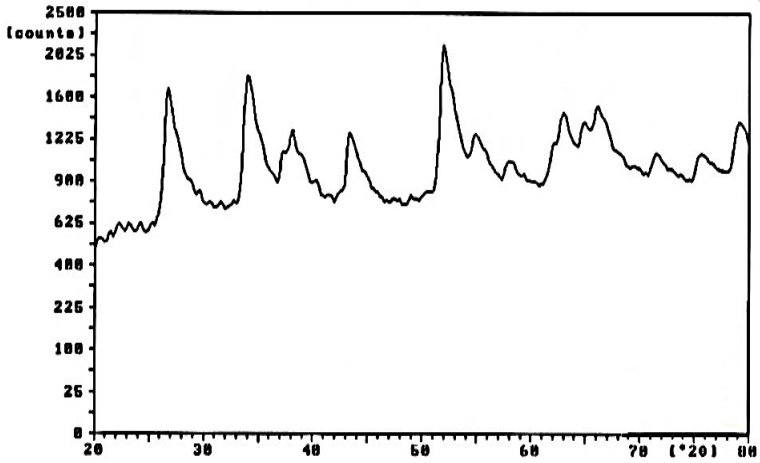
$$Z = \frac{NV\rho}{M}$$

where M molecular weight of the material, N Avogadro number, V basic cell measured one size (cm) ^{3} [11].
Thus we find that:

$$Z=\frac{NV\rho}{M} = 6.013 \approx 6$$

Following the method of rounding we found out that Z = 6, and therefore we can write the general formula for the content of the basic cell as follows: $Ni_6Sn_6O_{18}$
It is necessary to point at the fact that the heating up of the compound Nickel stanate to a higher degree of 800°C leads to the disintegration of the raw materials as shown (Figure 3).

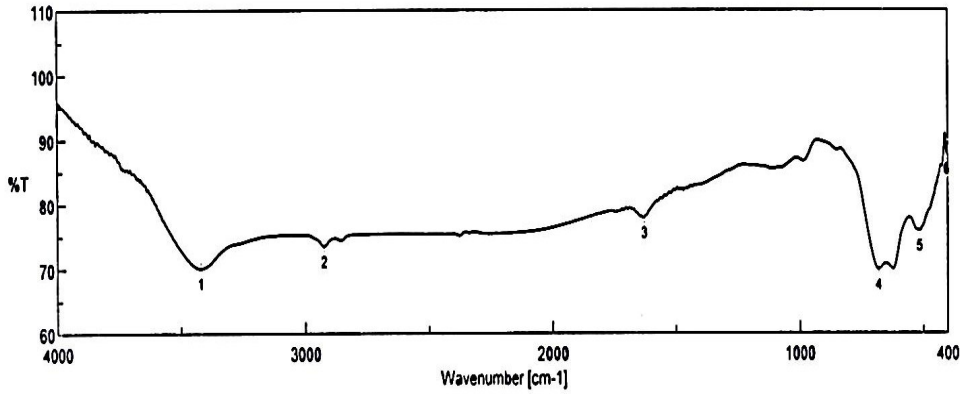
Figure 3. x-ray diffraction of the compound



resulting $NiSnO_3$ scheme (T = 800°C)

4.2.2 The study of compounds generated using the infrared spectrum:
It is very necessary to use your infrared spectrum infrared rays, and these rays are located in the area between the red rays in the visible rays, and a short-wave (microwave).

Is a spectral analysis of the absorption of IR radiation from the basic methods used in the automatic qualitative analysis through which to identify the installation of molecules and determine the functional cliques and chemical bonding and the quality of vibration, it can also be used to detect changes that are occurring molecules as a result of their interaction and the formation of new molecules.
The Figure 4 shows the radiation spectrum infrared sample Nickel stanate burned at degree 700°C.



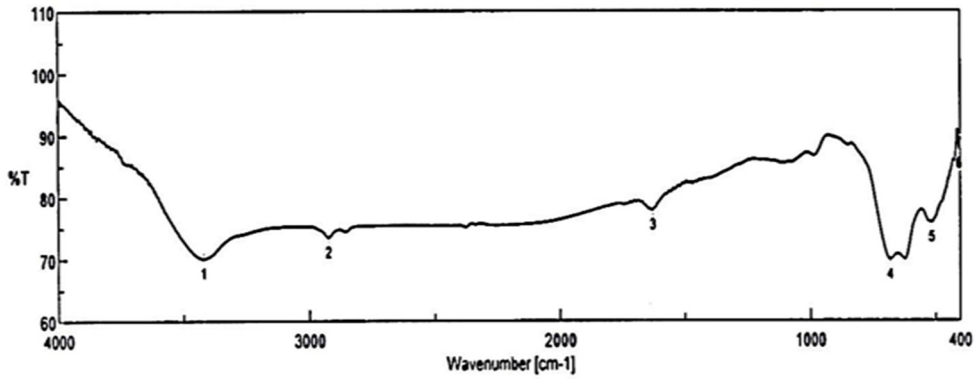


Figure 4. infrared spectrum IR compound $NiSnO_3$.

Where it appears from the spectrum that there are four-absorption values, the first absorption at the value 3424 Cm^{-1}, which is due to vibration stretching of the pound OH and the second at the value 530 Cm^{-1}, which is due to vibration stretching of the pound Ni-O, and the third has nearly equal to 680 Cm^{-1}, which goes back to vibration stretching of the pound Sn-O. It was the adoption of the reference number [6] spectrum analysis

The values that indicate the presence of links Ni-O and Sn-O and the presence of O-H bond due to the presence of some moisture, also confirm the presence of the compound required a $NiSnO_3$.

The study of the thermal behavior of the resulting compound gave the following curve, which includes four absorption peaks
(Endothermal effects). See Figure 5.

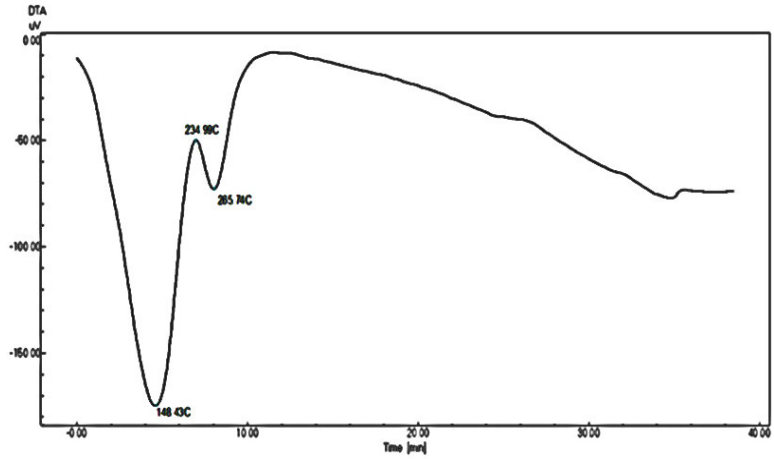


Figure 5.curved DTA differential thermal analysis
of the compound $NiSnO_3$.

The effect endothermic at degree 148.43°C indicating a loss of water molecules that are absorbed from the surrounding medium experience, and the thermal effect at the degree 265.99°C shows the composite $NiSnO_3$ turned into a developed, noncrystallised amorphous.

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